94-1 Core Technology Corrosion Research

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94-1 Core Technology Budget

- How this work fits into 94-1 Core Technology:
  - Materials ID
  - Stabilization
  - Packaging
  - Storage
  - Surveillance
- Customer: All sites where material will be packaged
- Current Budget $354k
  - NMT-15 (aqueous corrosion, SCC, Ga)
  - MST-6 atmospheric corrosion testing
  - University California Berkeley (SCC)
- Work is on schedule and on budget

FY 00 94-1 Core Technology Corrosion

3013 Container Activities:

- Gallium-induced failure
- Chloride-induced failure
- Shelf-life tests

Stress Corrosion Cracking of 3013 Containers (UCB):

- Conclusion of work 4/30/00

Complex-wide Consulting :

- MIS working group
- 3013 Standard
- Container failure at SRTC

Corrosion Milestones / Deliverables

- Submit at least one refereed journal article (9/30/00)
- Determine atmospheric conditions within container which result in localized corrosion
  - Design, build, operate atmospheric chamber (1/31/00)
  - Perform tests using different dewpoint / temperature combinations (9/30/00)
  - Report on results (MIS meetings) and author report at finish (FY 01?)
- Establish effect of Ga on LME susceptibility of containers
  - Make servohydraulic operable (11/30/00)
  - Establish testing procedure (3/31/00)
  - Report on results (MIS meetings) and author report at finish (FY 01?)
- Shelf-life corrosion studies
  - Design, build, calibrate atmospheric corrosion rate monitors (1/31/00)
  - Perform corrosion rate studies on shelf-life packages (ongoing)
  - Report on results (MIS meetings) and author report at finish (?)

Corrosion Milestones / Deliverables

- Assess RFETS laser welds
  - Compare corrosion performance of welded and as-received container materials (3/31/00)
  - Assess sensitization level of welds (3/31/00)
  - Compare stress corrosion cracking performance of welded and as-received container materials (9/30/00)
  - Report on results (MIS meetings) and author report (9/30/00)
- Examine the effect of stress on SCC of 316 SS
  - Produce thin film samples (4/30/00)
  - Test samples using concurrent SERS and mechanical testing (4/30/00)
  - Report on results in M.S. thesis and refereed journal articles (4/30/00)

Background

- The problem: There is concern that storage containers will not be suitable for 50 year storage of Pu compounds.

- DOE-STD-3013-99 states that containers incorporate 30 wt% to 100 wt% Pu (< 19W). The containers may also incorporate:
  - Ga bearing compounds from weapons Pu
  - Water (up to **0.5 wt%**) / H_2 / O_2
  - Salts / chlorinated compounds
  - Elevated temperature (Maximum excursion Temperature = **250^{o}C**)
  - Ionizing radiation
  - Welds (inner and outer containers)

- Expected container materials:
  - Outer: 316L "Pressure Vessel". 1 or 2 welds.
  - Inner: 316 SS. Laser cut and laser welded.
  - Convenience container (for metal): 304L body, 416 lid.
  - Convenience container (for oxide): 316 body, 416 lid.

Background

❖ Complex-wide experience suggests that significant problems will not occur. However, not all compounds have been stored and stored compounds have not necessarily experienced expected thermal conditions.

❖ Out of 113 RFETS containers, only four have shown evidence of corrosion attack.

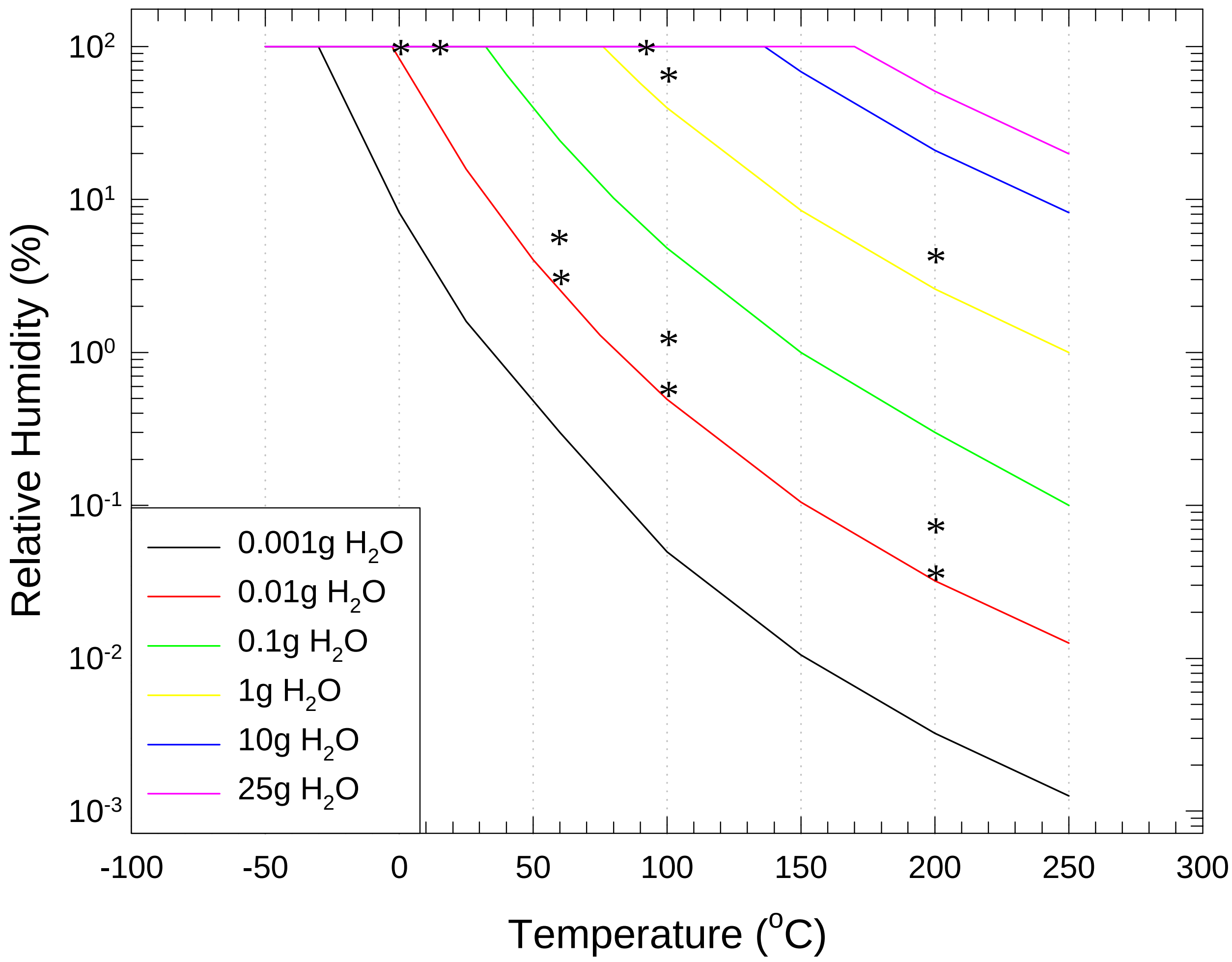
Can No.	Packaging Date	Source of Salt	Moisture	Corrosion
D17290	4/17/80	MSE	1.5%	Rust
D77584	9/30/91	ER	1.6%	Rust
2904061	4/25/88	DOR	1.8%	Slight Rust
D37748	9/30/83	DOR	0.6%	Slight Rust

3013 Containers - Potential Failure Mechanisms

- Corrosion:
  - Ga
  - Cl
  - H_2O
  - H_2O Radiolysis Products
  - Molten Salt
  - Sensitization (Thermal or Radiation-Induced)
  - Weld
- Environmental Cracking:
  - "Conventional" H_2O / O_2 / Cl^- / Cl_2 / HCl
  - Radiolysis
  - Welds
  - Ga Metal Embrittlement
  - Molten Salt
  - Sensitization (Thermal or Radiation-Induced)
- Embrittlement:
  - Radiation
  - H_2
  - Weld
  - Transmutation
- Alloying:
  - Ga / Fe
  - Pu / Fe

Absolute Corrosion Susceptibility

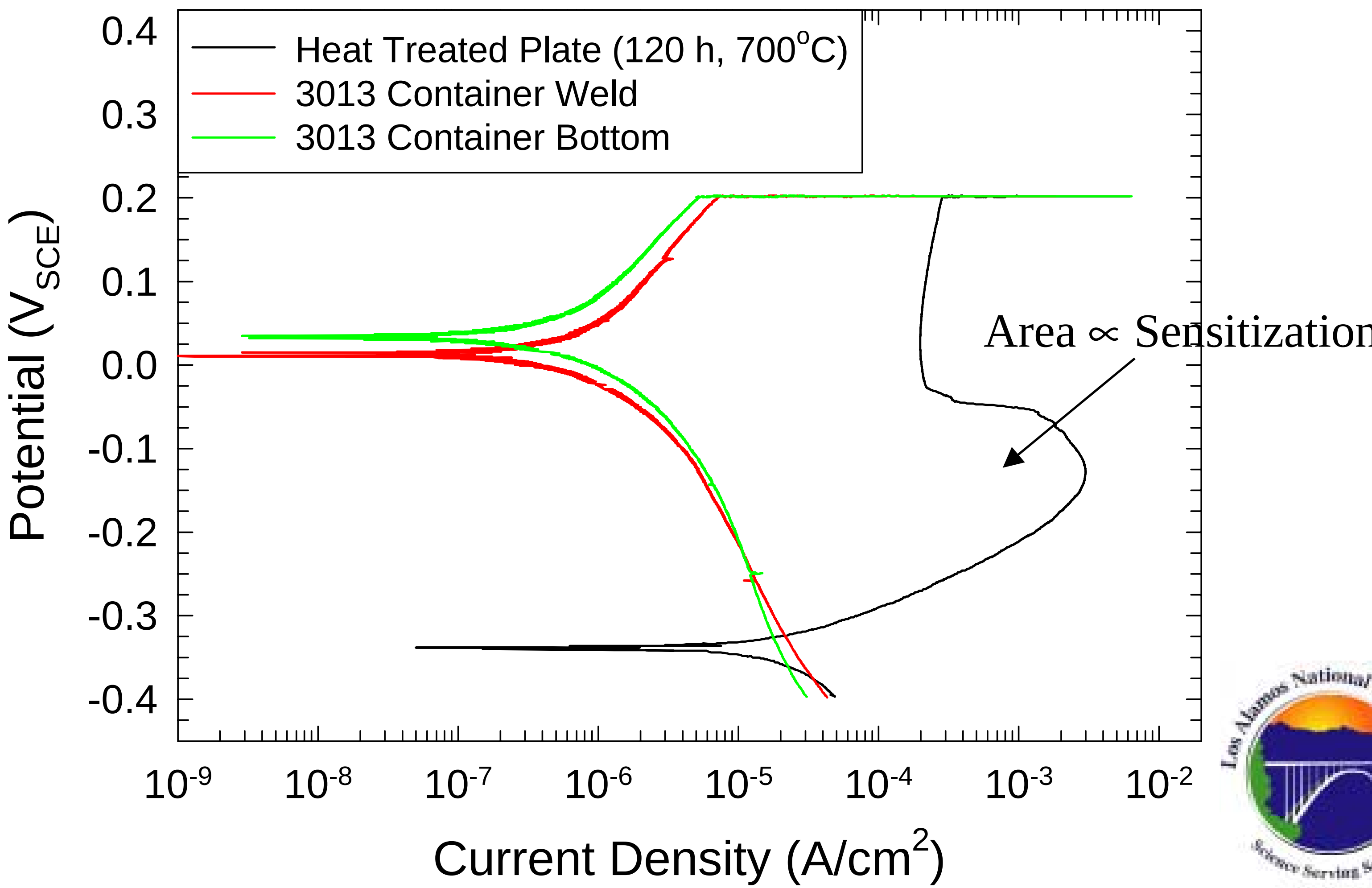
- Assuming:
  - All water is **free** to vaporize
  - 2700 cm^3 interior volume
  - LANL atmospheric pressure upon packaging



Laser & TIG Weld Sensitization

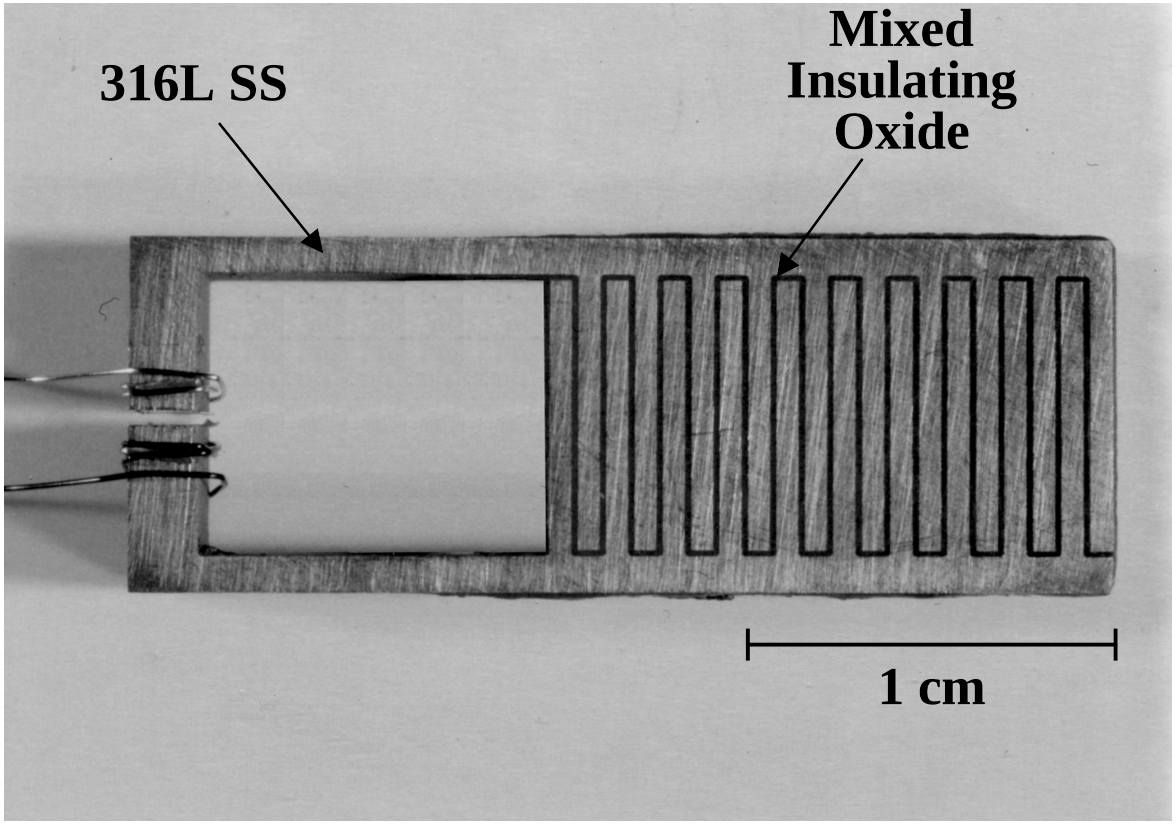
❖ Determine sensitization level of of RFETS laser weld using a standard test (SLEPR)

316 SS, 30ºC, 0.5 M H_2SO_4 + 0.01 M KSCN



Shelf-Life: In-Situ Corrosion Monitoring

- Atmospheric corrosion rate monitor (ACRM)
  - Linear Polarization Resistance
  - Electrochemical Impedance Spectroscopy



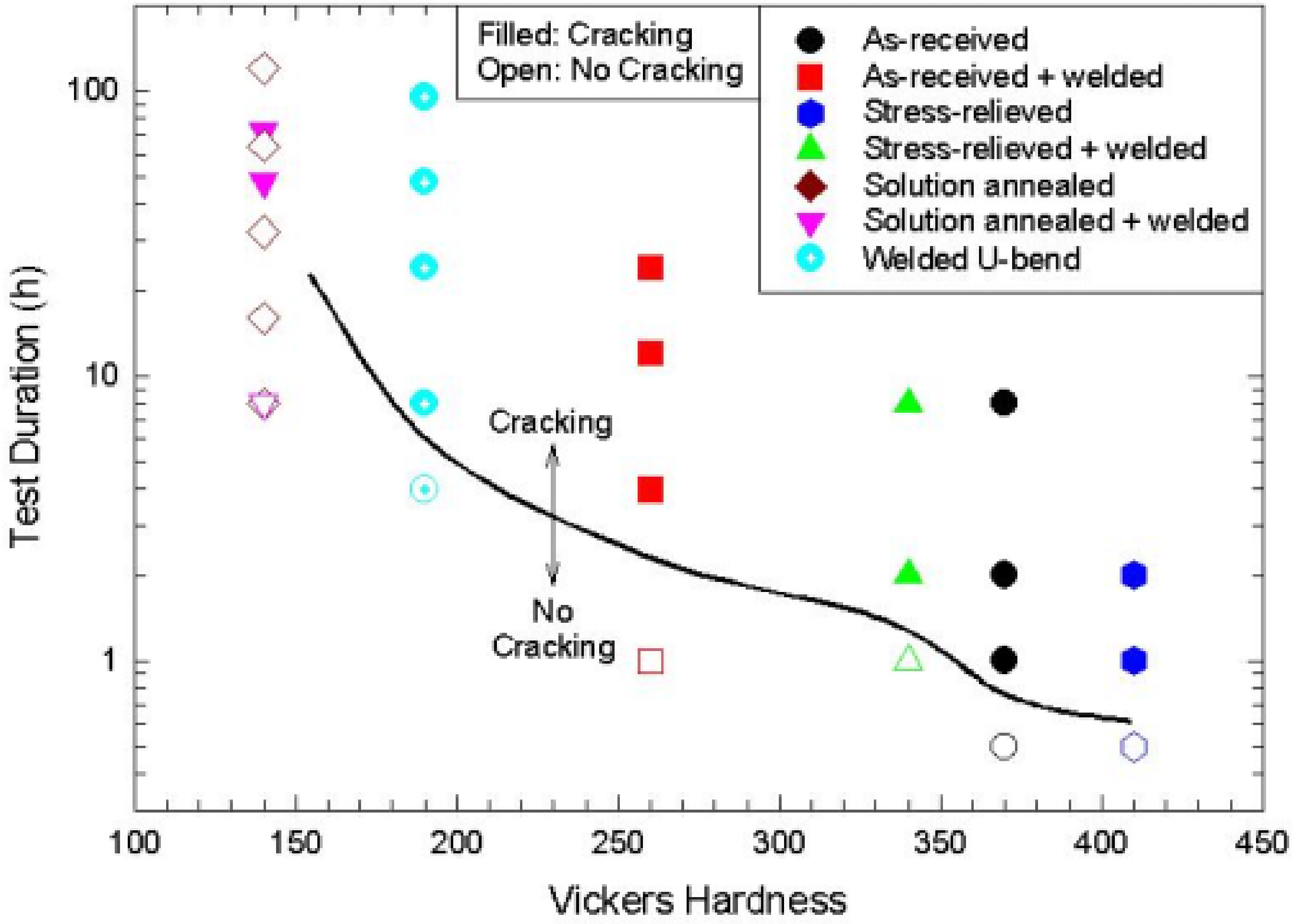
Stress Corrosion Cracking

- All of the components required for stress corrosion cracking of austenitic stainless steels are present:
  - Water (vapor and possibly liquid)
  - Chloride
  - Oxygen / Peroxide
  - Stress

- Mitigated by:
  - Reducing water concentration
  - Reducing oxygen concentration
  - Reducing stress (residual and applied)
  - Avoiding dewpoint

Relative Susceptibility to Cracking (ASTM G36)

- ❖ No apparent sensitization following TIG welding

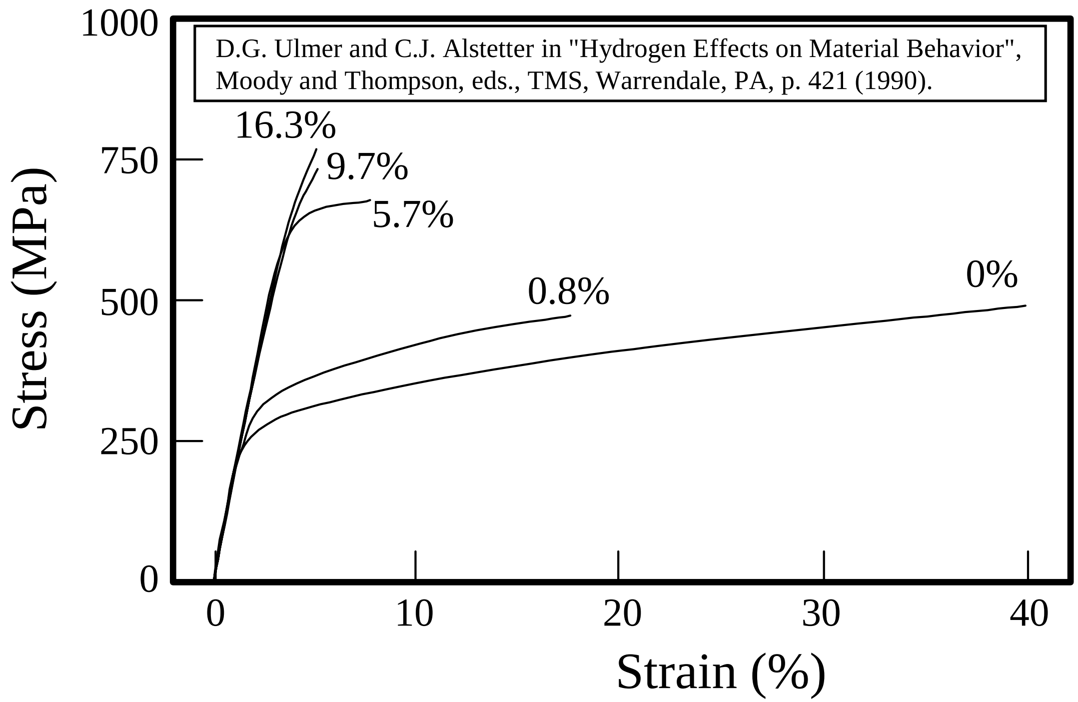


316 SS
As-received
8 h

Embrittlement of SS

❖ Hydrogen Embrittlement

✧ Requires significant plastic deformation for H absorption. Therefore, H embrittlement is not a concern.

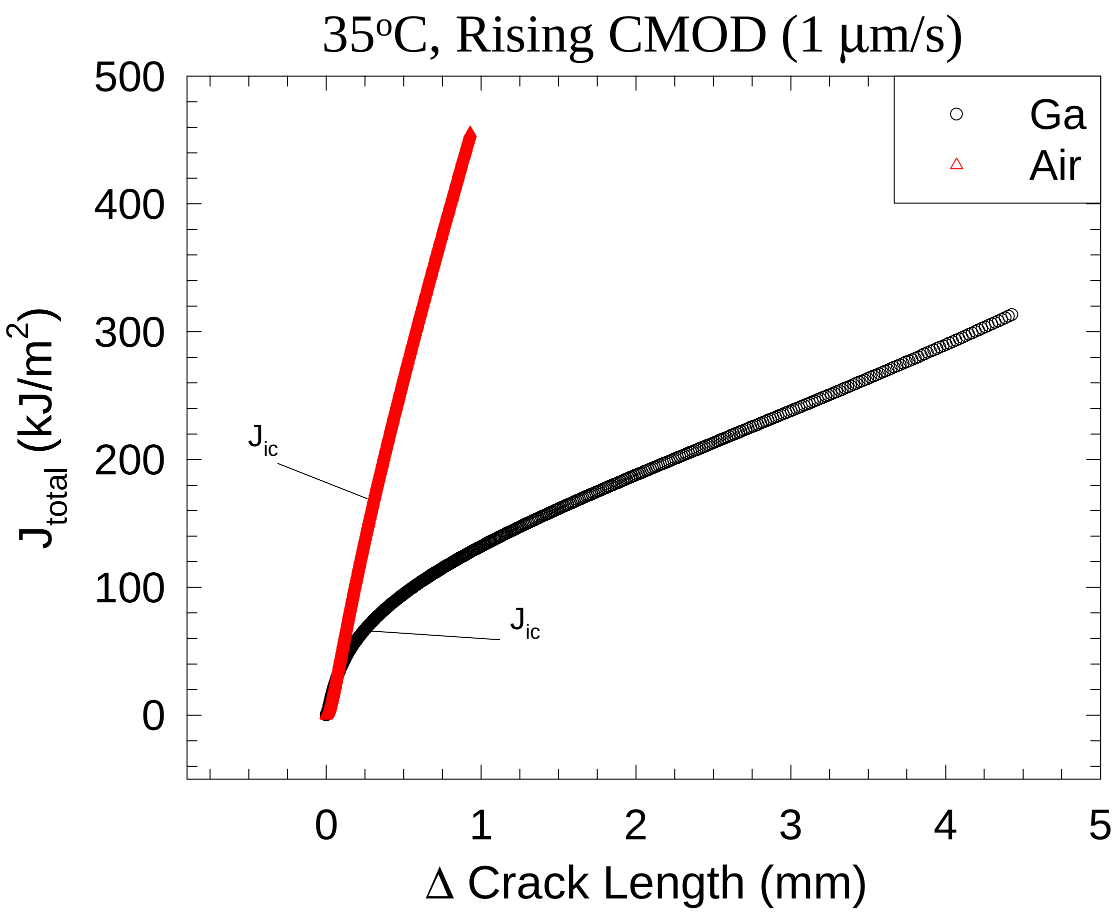


❖ Radiation Embrittlement

✧ Requires fluxes 10,000 times higher than that estimated over a 50 year lifetime. Therefore, radiation embrittlement is not a concern.

Environmental Cracking Liquid Metal Ga

- Gallium-induced cracking
  - Preliminary tests @ 35°C indicate that liquid metal Ga embrittles 316 L SS.
  - Unstable crack growth is not observed.
  - Requires Ga wetting and reduction of Ga_2O_3 in the case of PuO_2.

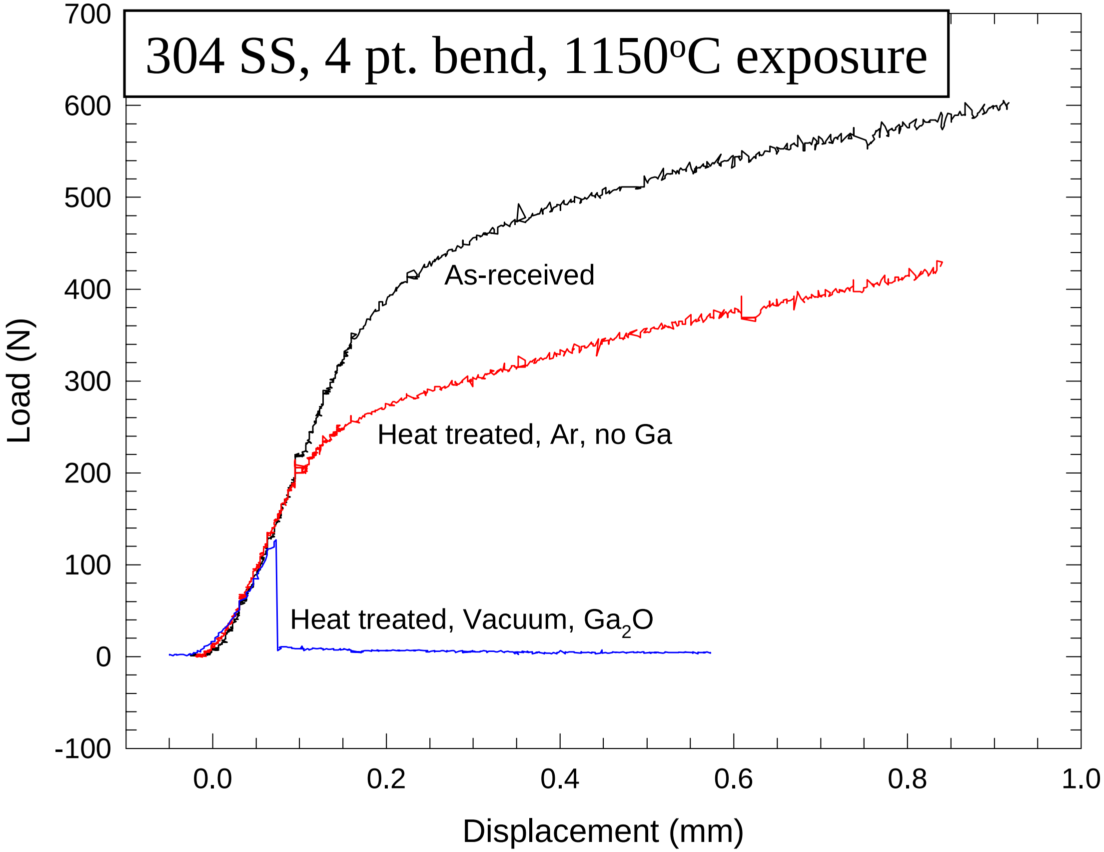


Alloying

- Pu - Fe
  - Based on analyses by Williamson Pu - Fe alloying is not expected to affect storage container integrity. (M.A. Williamson, "Plutonium Storage: Phase Equilibria Issues", Los Alamos National Laboratory Report # LA-UR-99-136, January 1999.)

- Fe - Ga
  - Embrittlement of 304 SS is observed in 22°C tests following Ga uptake (30 - 50 wt%) at 1150°C (in Ga_2O). Significant alloying of 316 SS does not appear to occur at temperatures $\leq$ 200°C (1000 h) but can occur at T $\geq$ 300°C. (L.R. Kelman et al., Argonne National Laboratory Report # ANL-4417 (1950) & P.R. Luebbers et al., Argonne National Laboratory Report # ANL-93/31, December 1993.

Ga Embrittlement



3013 Containers - Potential Failure Mechanisms

- Corrosion:
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  - Cl
  - H_2O
  - H_2O Radiolysis Products
  - Molten Salt
  - Sensitization (Thermal or Radiation-Induced)
  - Weld
- Environmental Cracking:
  - "Conventional" S.C.C.: H_2O / O_2 / Cl^- / Cl_2 / HCl
  - Radiolysis
  - Welds
  - Ga Metal Embrittlement
  - Molten Salt
  - Sensitization (Thermal or Radiation-Induced)
- Embrittlement:
  - Radiation
  - H_2
  - Weld
  - Transmutation
- Alloying:
  - Ga / Fe
  - Pu / Fe

Conclusions-To-Date

- ## Corrosion:

  ✧ Experiment: Corrosion per se does not appear to be a concern. Greatest current concern is the ability of corrosion pits to nucleate stress corrosion cracks

  ✧ Experiment: Corrosion and SCC of TIG welds does not appear to be a problem.

- ## Embrittlement:

  ✧ Literature: Radiation flux is too low for radiation embrittlement

  ✧ Literature: Lack of significant plastic deformation obviates hydrogen embrittlement concerns

  ✧ Experiment: Ga embrittlement occurs at 35^{o}C

Conclusions-To-Date

- Alloying:
  - Literature: Pu – Fe and Ga – Fe alloying does not appear to be a concern

- Stress Corrosion Cracking:
  - Experiment: TIG welds do not have largest effect on SCC susceptibility. Largest effects are carbon content and residual stress.
  - SCC presents the greatest threat to container integrity. All of ingredients for SCC are present and only a small amount of reactant is needed for failure. The elimination of residual stress and water is critical to avoid SCC failure.